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Indian Standard
SPECIFICATION FOR STABILIZED LIQUID SULPHUR TRIOXIDE

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110001

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Indian Standard
SPECIFICATION FOR STABILIZED LIQUID SULPHUR TRIOXIDE

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Indian Standard
SPECIFICATION FOR STABILIZED LIQUID SULPHUR TRIOXIDE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 December 1974, after the draft finalized by the Acids and Fertilizers Sectional Committee had been approved by the Chemical Division Council.

0.2 Stabilized liquid sulphur trioxide is widely used as sulphonating agent in chemical industry. It solidifies at or below 16.8°C.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for stabilized liquid sulphur trioxide, used as sulphonating agent in chemical industry.

2. REQUIREMENTS

2.1 Description — The material shall be a transparent liquid. It shall be colourless or have a brownish tinge, free from suspended matter and visible impurities.

2.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A. References to the relevant clauses of the appendix are given in col 4 of the table.

3. PACKING AND MARKING

3.1 Packing

3.1.1 The material is generally packed in mild steel tankers, insulated if

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*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR STABILIZED LIQUID SULPHUR TRIOXIDE

(Clause 2.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO Cl No. OF APPENDIX A)
(1)	(2)	(3)	(4)
i)	Free sulphur trioxide, percent by mass, *Min*	99.2	A-2
ii)	Sulphuric acid (as H_2SO_4), percent by mass, *Max*	0.4	A-3
iii)	Iron (as Fe), parts per million, *Max*	100	A-4
iv)	Oxidizable impurities (as SO_2), percent by mass, *Max*	0.2	A-5

necessary. Other packings may be used if agreed to between the manufacturer and the purchaser, subject to the provisions of Red Tariff No. 18 of 1960 issued by the Indian Railways Conference Association, with any alteration or addition made thereafter.

3.1.2 When the material is supplied in ground glass bottles the stoppers shall be secured with a metal wire. The stoppers shall further be sealed by a putty made of china clay or a mixture of sodium silicate and asbestos flour.

3.1.3 The bottles and jars shall be packed in suitable pent top packing cases. These shall be placed in an upright position on one layer of sand, or ashes free from cinders, or dry earth, and the empty surrounding space shall also be filled with the same material to prevent movement.

3.1.3.1 The material shall be stored between 17 and 30°C.

3.2 Marking

3.2.1 The containers and also the packages shall be suitably marked in red letters not less than 2.5 cm high, and drums and tank wagons shall be marked in red letters not less than 5 cm high, showing the name of the material, manufacturer's name, the mass of the material, recognized trade-mark, if any, and the month and the year of manufacture; and shall prominently display the words:

'CORROSIVE, HANDLE WITH CARE'

3.2.2 The packages shall be marked with the percent content (m/m) of the stabilizer and, if the stabilizer is acidic to methyl red indicator, the volume of 0.5 N sodium hydroxide solution required to neutralize the stabilizer present in 100 g of the material.

3.2.3 The packages shall be labelled as shown in Fig. 1.

3.2.4 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark is granted to manufacturers or processors may be obtained from the Indian Standards Institution.

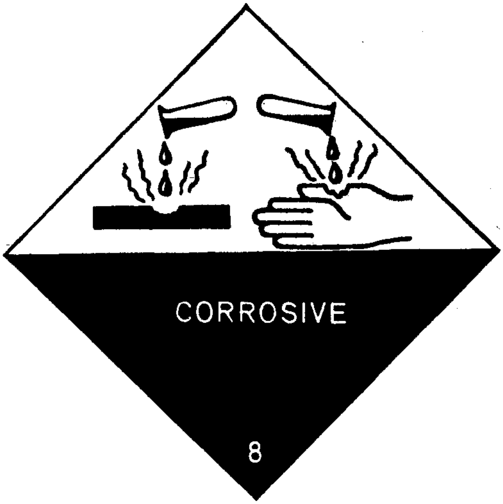

FIG. 1 SYMBOL SHOWING DANGER OF CORROSION

4. SAMPLING

4.1 Representative test samples of the material shall be drawn as prescribed in Appendix B.

APPENDIX A

(Clause 2.2)

METHODS OF TEST FOR STABILIZED LIQUID SULPHUR TRIOXIDE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water *(see* IS : 1070-1960*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF FREE SULPHUR TRIOXIDE

A-2.1 Apparatus—The apparatus prescribed under **A-2.1.1** to **A-2.1.5** shall be fitted with suitable ground glass joints *(see* IS : 5165-1969†).

A-2.1.1 *Flask* — 125 ml capacity, provided with a stopper.

A-2.1.2 *Connecting Tube* — in the shape of an inverted U-tube.

A-2.1.3 *Water Condenser — See* IS : 6052-1970‡.

A-2.1.4 *Glass Adaptor*

A-2.1.5 *Receiver Flask* — with a narrow neck for collecting liquid sulphur trioxide.

A-2.1.6 *Heating Mantle* — a hemispherical heating mantle to fit the flask.

A-2.1.7 *Variable Transformer* — to adjust the known input into the heating mantle.

A-2.2 Procedure

A-2.2.1 Add 3 to 4 glass beads to the thoroughly dry flask, stopper the flask, and weigh. Remove the stopper and insert the funnel in the flask. Pour about 100 g of the sample into the flask. Remove the funnel, stopper the flask, and weigh it. Connect the flask by means of the connecting tube to the water condenser and place the receiver under the end of the adaptor to collect liquid sulphur trioxide. Place the flask in the heating mantle and adjust the temperature (about 45° to 50°C) so that the sample boils gently. Do not attempt to heat on a steam or water-bath.

A-2.2.2 When evolution of sulphur trioxide fumes ceases, cool the flask, remove the connecting tube and quickly blow out of the flask the sulphur trioxide vapours with a stream of thoroughly dried air. Dry the inside of the neck of the flask with a stream of thoroughly dried air; restopper with the

---

*Specification for water, distilled quality *(revised).*

†Specification for interchangeable conical ground glass joints.

‡Specification for glass condensers.

original stopper and weigh the residue. Retain the stoppered flask with the residue for test in **A-3.**

A-2.3 Calculation

$$\text{Free sulphur trioxide, percent by mass} = \frac{100\,(M_2 - M_1)}{M_2}$$

where

M_2 = mass in g of the material taken for the test, and
M_1 = mass in g of the residue.

A-3. DETERMINATION OF SULPHURIC ACID

A-3.1 Reagents

A-3.1.1 *Standard Sodium Hydroxide Solution* — 0.5 N.

A-3.1.2 *Methyl Red Indicator* — Dissolve 0.1 g of methyl red in 100 ml of rectified spirit.

A-3.2 Procedure

A-3.2.1 Carefully add about 30 ml of water to the stoppered flask containing the residue (*see* **A-2.2.2**) and allow to stand until all mist has been absorbed. Transfer the solution to a 100-ml volumetric flask and make the volume up to the mark by washings of the flask. Use this solution for tests given in **A-3.2.2, A-4.1** and **A-4.2.**

A-3.2.2 Take 20 ml of the solution in a flask with the help of a pipette. Titrate the solution against standard sodium hydroxide solution using methyl red as indicator. Apply a correction for the volume of sodium hydroxide solution used by the stabilizer.

A-3.3 Calculation

$$\text{Sulphuric acid (as } H_2SO_4\text{), percent by mass} = \frac{4{\cdot}9\,(5\,N\,V - 0{\cdot}5\,V_1)}{M}$$

where

N = normality of standard sodium hydroxide solution,

V = volume in ml of standard sodium hydroxide solution used in the titration in **A-3.2.2,**

V_1 = volume in ml of standard sodium hydroxide of normality 0.5 N that would be consumed by the stabilizer present in M g of the sample (*see* **3.2.2**), and

M = mass in g of the sample taken for the test in **A-2.2.**

A-4. DETERMINATION OF IRON

A-4.0 General — Two methods are prescribed. Method A is the referee method and Method B is the alternate method.

A-4.1 Method A

A-4.1.1 *Apparatus*

A-4.1.1.1 *Nessler tubes* — 50 ml capacity.

A-4.1.2 *Reagents*

A-4.1.2.1 *Ammonium persulphate*

A-4.1.2.2 *Concentrated hydrochloric acid*

A-4.1.2.3 *Butanolic potassium thiocyanate solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient n-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-4.1.2.4 *Standard iron solution A* — Weigh, to the nearest 1 mg, 7.022 g of iron (II) ammonium sulphate hexahydrate and place in a beaker of suitable capacity. Add 50 ml of sulphuric acid (about 2 N) and transfer quantitatively to a 500-ml one-mark volumetric flask. Dilute to the mark and mix thoroughly. One millilitre of this solution contains 200 mg of iron as Fe.

A-4.1.2.5 *Standard iron solution B* — Transfer 500 ml of the standard iron solution *A* to a 500-ml one-mark volumetric flask, add 5 ml of sulphuric acid (about 2 N), dilute to the mark and mix thoroughly. One millilitre of this solution contains 0.20 mg of iron as Fe. The solution should be prepared just before use.

A-4.1.3 *Procedure*

A-4.1.3.1 Pipette out 10 ml of the solution reserved in **A-3.2.1,** into a Nessler tube. Add about 30 mg of ammonium persulphate, 1 ml of concentrated hydrochloric acid and 15 ml of butanolic potassium thiocyanate solution. Make up to 50 ml, shake vigorously for 30 seconds and allow the layers to separate. Carry out a control test in another Nessler tube in the same manner using 5 ml of standard iron solution *B* and compare the intensity of colour produced in the butanol layers in the two tubes.

A-4.1.3.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the red colour produced in the butanol layer with the sample is not greater than that produced in the control test.

A-4.2 Method B

A-4.2.1 *Apparatus*

A-4.2.1.1 *Spectrophotometer or photoelectric colorimeter*

A-4.2.2 *Reagents*

A-4.2.2.1 *Hydrochloric acid* — approximately 1 N.

A-4.2.2.2 *Hydroxylammonium chloride solution* — Dissolve 10 g of hydroxylammonium chloride in water and dilute to 100 ml.

A-4.2.2.3 *Ammonium acetate solution* — Dissolve 30 g of ammonium acetate in water and dilute to 100 ml.

A-4.2.2.4 *Bipyridyl solution* — Dissolve 1 g of 2, 2'-bipyridyl in 10 ml of approximately 1 N hydrochloric acid solution and dilute to 100 ml.

A-4.2.2.5 *Standard iron solution C* — Transfer 50.0 ml of standard iron solution *B* to a 1 000-ml one-mark volumetric flask, dilute to the mark and mix thoroughly. One millilitre of this solution contains 10 µg of Fe. The solution should be prepared just before use.

A-4.2.3 *Procedure*

A-4.2.3.1 *Preparation of calibration curve* — Into each of a series of 100 ml one-mark volumetric flasks, place the quantities of standard iron solution *C* indicated below:

Standard Iron Solution C (ml)	*Corresponding to Fe* (µg)
0 (Compensation solution)	0
5.0	50
7.5	75
10.0	100
12.5	125
15.0	150
17.5	175
20.0	200
25.0	250

Add to each volumetric flask an amount of water sufficient to dilute to approximately 50 ml, then 2 ml of hydrochloric acid solution, 2 ml of hydroxylammonium chloride solution and after 5 minutes, 5 ml of ammonium acetate solution and 1 ml of the 2, 2'-bipyridyl solution. Dilute to the mark, mix thoroughly and wait for 10 minutes. Carry out the measurement in the spectrophotometer in a 1 cm cell at a wavelength of about 522 mm, or in a photoelectric colorimeter using an appropriate filter, adjusting the instrument to zero optical density using as reference the compensation solution. Prepare a calibration chart having for example the iron content in micrograms per 100 ml of the standard matching solution as abscissae and the corresponding values of optical density as ordinates.

A-4.2.3.2 Transfer quantitatively to a 100 ml one-mark volumetric flask 20 ml of solution prepared in **A-3.2.1** and 2 ml of hydrochloric acid, dilute to the mark, mix and filter if necessary. Transfer an aliquot of the sample solution containing between 50 and 200 µg of iron, to a 100 ml onemark volumetric flask. Dilute to approximately 50 ml if necessary, then add successively 2 ml of hydrochloric acid solution, 2 ml of the hydroxylammonium chloride solution and, after 5 minutes, 5 ml of ammonium acetate solution and 1 ml of 2, 2'-bipyridyl solution. Dilute to the mark, mix and wait for 10 minutes. Carry out the spectrophotometric measurement according to the procedure given in **A-4.2.3.1** adjusting the instrument to zero optical density using as reference the blank test solution *(see* **A-4.2.3.3**). Alternatively, colour may also be compared using a photoelectric colorimeter.

A-4.2.3.3 *Blank test* — At the same time as the analysis, carry out a blank test using the same procedure and quantities of all reagents employed in the test.

A-4.2.4 *Calculation* — By reference to the calibration chart *(see* **A-4.2.3.1)** determine the iron content corresponding to the spectrophotometric measurement.

The iron content is calculated by the following formula:

$$\text{Iron (as Fe), percent by mass} = \frac{A \times 100 \times 100}{V \times M}$$

where

A = mass in g of iron determined in the aliquot of the sample solution,

V = volume in millilitres of the sample solution taken for the colour reaction, and

M = mass in g of the test portion **(A-2.2).**

A-5. DETERMINATION OF OXIDIZABLE IMPURITIES

A-5.1 Apparatus

A-5.1.1 *Flask* — 125 ml capacity, with a stopper.

A-5.1.2 *Ice Bath* — a porcelain dish with ice cold water.

A-5.2 Reagents

A-5.2.1 *Standard Potassium Permanganate Solution* — 0.1 N.

A-5.3 Procedure — Weigh the dry flask with stopper. Remove the stopper, insert a funnel in the flask, and pour about 50 g of the sample into the flask. Stopper the flask and weigh again. Cool the flask in ice-cooled water for half an hour, and then replace the original stopper with a bored rubber cork. Insert the tip of a burette containing standard potassium permanganate solution through the bore of the cork into the flask, keeping it in ice-cooled water-bath. Run the permanganate solution from the burette into the flask drop by drop shaking the contents of the flask vigorously during the titration to avoid spurting of liquid sulphur trioxide. The end point is seen visually when the solution acquires a slight pink colour.

A-5.4 Calculation

$$\text{Oxidizable impurities (as } SO_2\text{), percent by mass} = \frac{3{\cdot}2 \times V \times N}{M}$$

where

V = volume in ml of standard permanganate solution used,
N = normality of standard permanganate solution, and
M = mass in g of the sample taken for the test.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF STABILIZED LIQUID SULPHUR TRIOXIDE

B-1. GENERAL REQUIREMENTS

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 The material shall be sampled in liquid condition.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples, from adventitious contamination.

B-1.6 The samples shall be placed in suitable, clean, dry and air-tight glass containers, on which the material has no action.

B-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.8 Each sample container shall be sealed airtight with a suitable stopper after filling, and marked with full details of sampling and the month and year of manufacture of the material.

B-1.9 Samples shall be stored in a dry place, between 17 and 30°C.

B-2. SCALE OF SAMPLING

B-2.1 From Containers or Packages

B-2.1.1 *Lot* — All containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately, and the groups of containers in each batch shall constitute separate lots.

B-2.1.2 Sample shall be tested from each lot separately for judging the conformity of the material to the requirements of the specification. For this purpose 5 containers shall be selected at random from each lot.

NOTE — If the number of containers in the lot is five or less, the number of containers to be selected and the criteria for conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

B-2.1.3 The containers shall be selected at random. In order to ensure randomness of selection, random sampling procedures given in IS : 4905-1968* may be followed.

B-2.2 From Tankers — Samples shall be taken from each tanker.

B-3. PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE

B-3.1 For Packages or Containers

B-3.1.1 *Sampling Device* — The sampling device shall consist of a glass bottle (sampling vessel) with the necessary connections as shown in Fig. 2. The connecting tubes shall be made of glass or teflon. The samples shall be drawn from the containers from the bottom.

B-3.1.2 From each of the containers selected according to **B-2.1.3,** a small representative portion of the material, about 300 ml, shall be taken out with the help of the sampling device after thoroughly stirring the material with the help of a glass rod.

B-3.1.3 Out of these portions, a small but equal quantity of the material shall be taken out and thoroughly mixed to form a composite sample not less than 1 000 ml. The composite sample shall be divided into 3 equal parts, one for the purchaser, one for the supplier and the third to be used as a referee sample.

B-3.1.4 The remaining portion of the material from each container shall be divided into 3 equal parts, each forming an individual sample. One set of individual samples representing the 5 containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.1.5 All the individual and composite samples shall be transferred to separate bottles. These bottles shall be sealed and labelled with full identification particulars.

B-3.1.6 The referee samples consisting of composite sample and a set of 5 individual samples shall bear the seals of purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier to be used in the case of a dispute between the two.

B-3.2 For Tankers — A sampling device similar to that prescribed in **B-3.1.1** and of suitable dimension shall be employed for taking out the

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*Methods for random sampling.

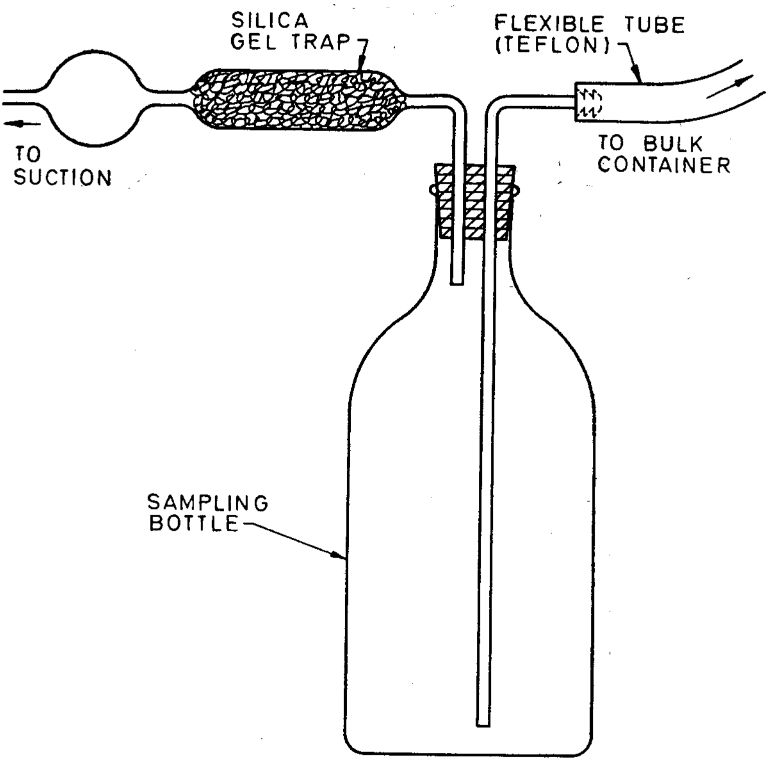

FIG. 2 SAMPLING INSTRUMENT FOR LIQUID SULPHUR TRIOXIDE, STABILIZED

samples. Samples shall be taken out from the bottom so that in all 1 litre is taken out. The material shall be thoroughly mixed and divided into 3 equal parts, one for the purchaser, another for the supplier and the third for the referee. These samples shall be transferred to separate bottles. The bottles shall be sealed and labelled with full identification particulars.

B-4. NUMBER OF TESTS

B-4.1 For Containers or Packages

B-4.1.1 Tests for the determination of free sulphur trioxide content shall be performed on each of 5 individual samples.

B-4.1.2 Tests for all characteristics given in **2** except free sulphur trioxide, shall be performed on the composite sample.

B-4.2 For Tankers — Tests for all the characteristics shall be carried out on the sample prepared for each tanker.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Containers or Packages

B-5.1.1 *For Individual Samples* — From the 5 test results for free sulphur trioxide content, the mean (x) and range (R) of test results shall be calculated (range being the difference between the maximum and minimum values of test results).

B-5.1.1.1 The lot shall be declared as conforming to the requirement for free sulphur trioxide if the value of the expression ($\bar{x} — 0.6\,R$) as calculated from the test results is greater than or equal to the minimum value specified in **2**.

B-5.1.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirements specified in **2.**

B-5.2 For Tankers — For declaring the conformity of the material in a tanker, the test result for each of the characteristics shall satisfy the requirements prescribed in **2.**

INDIAN STANDARDS

ON

MINERAL ACIDS

IS

264-1968 Nitric acid (*first revision*)
265-1962 Hydrochloric acid (*revised)*
265-1961 Sulphuric acid (revised)
798-1968 other phesphoric acid (*first revision*)
1089-1973 Oleurn. technical (*first revision*)
7541-1974 Stabilized liquid sulphur trioxide

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